準安定 β 型チタン合金の硬度に及ぼす微細組織の影響[1]

Effect of Microstructure on Hardness in Metastable β Titanium Alloy

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Microstructural evolution and variation of Vickers hardness (Hv) during aging at 673 K in a metastable β Ti alloy have been investigated. Value of Hv increases with increasing aging time (t). The value of Hv after aging for 37.5 ks is 498. After solution treatment followed by quenching into iced brine, athermal ω phase forms. The athermal ω phase is replaced into isothermal one during aging. The isothermal ω phase acts as nucleation site for α precipitate, and the number density (ρ) and size of precipitates increases with increasing t. The continuous incresase in Hv is due to the nucleation of α precipitate at ω precipitate. The decrease of distance (d) between precipitates with increasing t results in the increase in Hv during aging.

Keywords: Metastable β titnanium alloy, Aging, Hardness, Transmission electron microscopy, High resolution electron microscopy

1. Introduction

Metastable β Ti alloys are widely used alloys owing to high specific strength for aerospace structures[1]. This kind of alloy contains β stabilizers enough to retain a high-temperature β phase (bcc) to room temperature metastably. After solution treatment, aging treatment is often applied to the alloys in order to enhance their strength and toughness by forming α precipitates (hcp)[2]-[4]. It has been tried to refine α precipitate through a lot of thermo-mechanical treatments[5]-[6]. As one of these treatments, two-step aging is often performed[7]. In two-step aging, the first aging is performed at a lower temperature where a fine precipitates of transient phase such as hexagonal ω phase appear, and then the second aging at a higher temperature where α phase nucleates at the transient phase.

Table 1 Chemical composition (mass%)

Al	Fe	Mo	O	N	H	Ti
1.54	4.59	7	0.148	0.011	0.0102	bal.

In order to lower the green house effect, it is necessary to lower the weight of cars, so Ti alloys are good candidates for automobile materials for high specific strength. TIMETAL®LCB is the alloy developed for automobile application to lower the cost of alloy[8]. This alloy consists of inexpensive elements, such as Fe, Mo and Al. In this alloy, ω phase precipitates as a transient phase[9], but there are few reports about α phase precipitation at the ω phase. In the present study, effect of microstructural evolution on hardness during aging at a temperature where ω and α phases form has been investigated.

2. Experimental Procedure

The alloy used was supplied from Titanium Metals Corporation (TIMET) and Nippon Steel Corporation. The chemical composition was listed in Table 1. Specimens were cut from the supplied bar, and subjected to solution treatment at 1173K for 1.5 ks, followed by quenching into iced brine. After that, the specimens were aged at 673K for 0.3 to 37.5 ks. Vickers microhardness was measured with weight of 300 g. Microstructure was observed by transmission electron microscopes (TEMs). TEMs used were JEM-100CXII, JEM-2000EX and JEM-3010 operated at 100, 200 and 300 kV, respectively. High resolution electron microscopy (HREM) was performed by JEM-3010. TEM specimens were prepared by mechanical grinding and twin-jet electropolishing at 100 V and 223 K. The electrolyte was 8%HClO$_4$, 32%C$_4$H$_9$OH and 60%CH$_3$OH in volume. Number density of precipitates, ρ, was measured by counting the number of precipitates in a dark field image and evaluating the volume of the relevant area. The specimen thickness, t, was evaluated from thickness fringe as follows; The value of t of a specimen in the region where the nth dark line of the fringe from the edge of the specimen is seen in a bright field image satisfies the following equation,

$$t = (n-1/2)\xi_{\text{eff}},$$

where ξ_{eff} is an effective extinction distance. The average distance between the precipitates, d, was evaluated from the following equation,

$$d = 1/\sqrt[3]{\rho}.$$

3. Results

3.1 Variations of Vickers Hardness duering Aging

Figure 1 shows Vickers microhardness (Hv) as a function of aging time (t). When a specimen was aged at 673K, Hv starts to increase after 0.3 ks, and after that, increases gradually to 498 for 37.5 ks. The Hv reaches 485 for 37.5 ks.

3.2 TEM Observation

Figure 2 shows TEM micrograph taken from solution-treated specimen. In the diffraction pattern, diffuse streaks derived from athermal ω phase are observed[9]. It was confirmed that athermal ω phase has Burgers orientation relationship with β matrix,
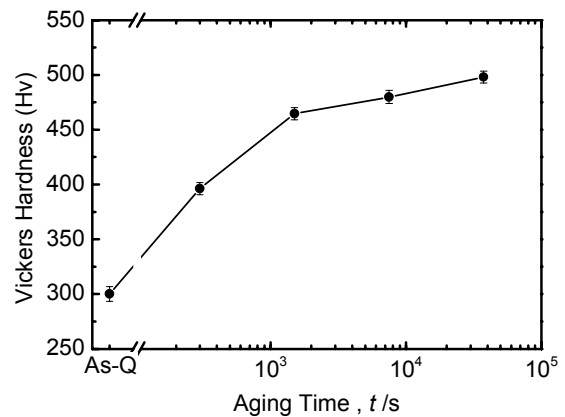

Fig. 1   Variation of Vickers microhardness with aging time.

$\{111\}_\beta // \{0001\}_\omega$ and $<1\bar{1}0>_\beta // <11\bar{2}0>_\omega$[10].

In the aged specimen at 673 K, isothermal ω phase is identified after aging for 0.3 ks as shown in Fig. 3(a). We confirmed this ω phase has also the same orientation relationship as that of athermal ω phase as shown in Fig. 2. The isothermal ω phase grows and ρ increases during aging as shown in Figs. 3(a)-(d). The ρ of the isothermal ω phase was measured. By using ρ, average distance between the isothermal ω precipitates, d, was evaluated as shown in Fig. 4. The value of d decreases with increasing t. In the specimen aged at 673 K for 37.5 ks, α phase precipitates as shown in Fig. 5. In this figure, the areas of the upper right corner were magnified in the insets. As shown in the insets, the α phase forms at the isothermal ω phase. This fact was also confirmed by HREM as shown in Fig. 6. In this figure, α phase forms at the interface between β and ω phases. Alpha, isothermal ω and β phases are in the following orientation relationship,

$$(1\bar{1}1)_\beta // (0001)_\omega // (\bar{1}\bar{1}20)_\alpha,$$
$$(\bar{1}01)_\beta // (\bar{1}\bar{1}20)_\omega // (0001)_\alpha \text{ and}$$
$$(\bar{1}2\bar{1})_\beta // (1\bar{1}00)_\omega // (\bar{1}010)_\alpha.$$

Alpha phase has Burgers orientation relationship[11] with β matrix. It is noted that α phase has an orientation relationship with ω phase as well.

4. Discussion

The value of Hv increases with increasing t during aging at 673 K as shown in Fig. 1. This increase in Hv is due to the decrease of d between isothermal ω precipitates as shown in Fig. 4. It is considered that dislocation motion is retarded by the ω precipitates. This result is in accordance with the theory of dispersion hardening, in which it is indicated that hardness increases with decreasing the spacing between dispersoids. After aging at 673 K for 37.5 ks, α phase forms, and Hv increases continuously as shown in Figs. 1 and 5. It is considered that the continuous increase in Hv
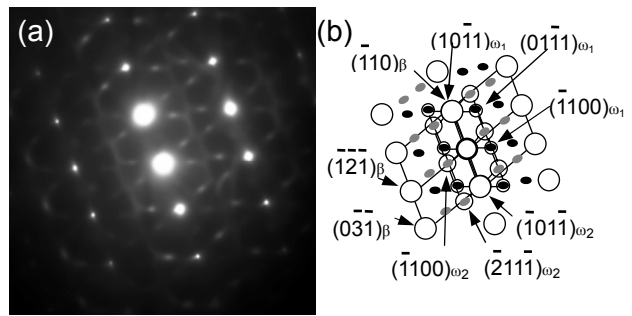

Fig. 2   Electron diffraction pattern taken from specimen solution-treated (a). (b) is the key diagram of (a).
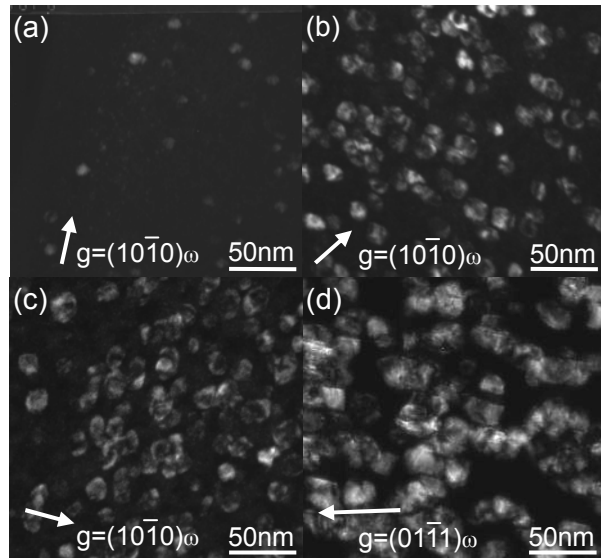

Fig. 3   Dark field images of isothermal ω phase aged at 673 K for (a) 0.3, (b) 1.5, (c) 7.5 and (d) 37.5 ks.
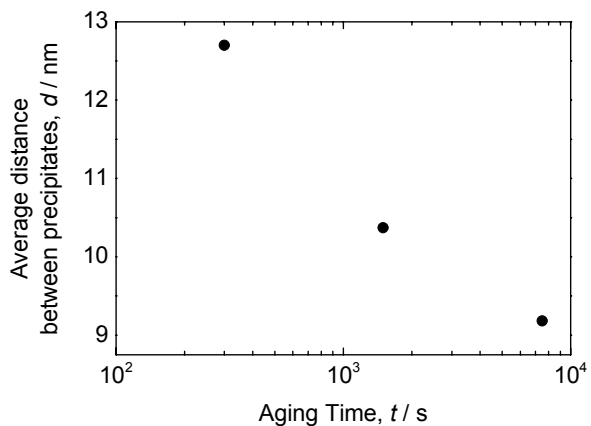

Fig. 4   Variation of average distance between isothermal ω precipitates with aging time.
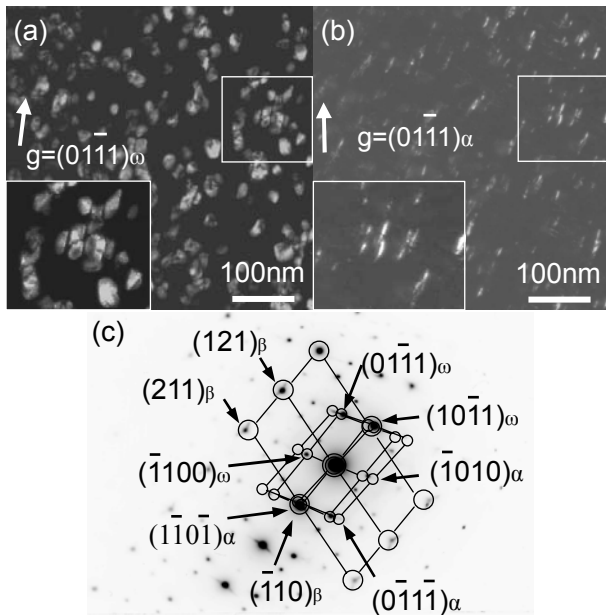
Fig. 5 Dark field images taken from (a) isothermal ω and (b) α phases in specimens aged at 673 K for 37.5 ks. Insets in (a) and (b) are magnified images of same area enclosed with white rectangles. (c) is the diffraction pattern.
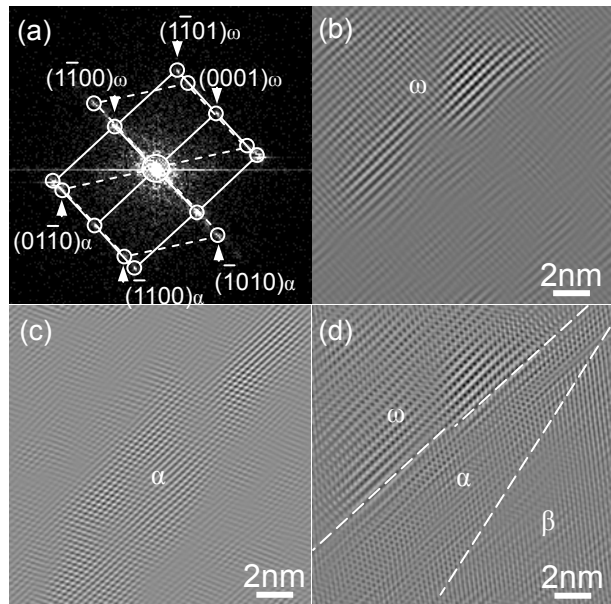
Fig. 6 (a) Fourier transform of HREM image taken from the specimen aged at 673 K for 37.5 ks. Inverse Fourier transform for (b) isothermal ω, (c) α   and (d) ω, α and β.

is because the value of ρ hardly changes owing to the nucleation of α phase at the interface between isothermal ω and β phases as shown in Fig. 6.

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6. References

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